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**Effect of Processing Schedule on the Microstructure and Texture of 0.78 wt% Cr Extra-
Low-Carbon Steel**

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Synopsis

An extra-low-carbon steel was alloyed with 0.78 wt% Cr and subjected to three different processing schedules involving: (i) warm rolling to 65% reduction at 640°C, (ii) warm rolling to 80% reduction at 580°C and (iii) warm rolling to 65% reduction at 640°C followed by cold rolling to 40% reduction. Increasing the severity of the deformation resulted in an increase in the number of grains containing in-grain shear bands. X-ray bulk texture analysis indicates that the γ -fibre intensity was slightly higher in the steel warm rolled at 580°C than at 640°C and after cold rolling. Transmission electron microscopy and atom probe tomography revealed that Cr carbides were formed after all processing schedules. Carbon segregation to dislocations was also observed. Although the addition of 0.78 wt% Cr produced a volume fraction of carbides that was three orders of magnitude higher than those previously observed in 0.48 wt% Cr steels, a significant amount of solute Cr remained confined in the matrix and did not lead to any further depletion of the solute carbon.

Keywords: extra-low carbon steel; warm rolling; atom probe tomography; texture; C segregation.

1.0 Introduction

In contrast to interstitial-free (IF) grades of steel which are essentially free of solute carbon, the presence of carbon in low-carbon steels leads to dynamic strain ageing during warm rolling at temperatures 300 - 700°C. This leads to the high strain rate sensitivity of the flow stress and consequently to a sharp reduction in the number of grains containing in-grain shear bands ¹⁻³). In turn, the lower in-grain shear band density is partly responsible for the weak but desirable normal direction (ND) texture (or γ fibre) that is produced upon annealing ¹). In order to reduce the effects of strain rate sensitivity, improve the bulk texture and formability and increase the volume fraction of in-grain shear bands, further addition of chromium (Cr) is regarded as a feasible approach during the warm (or ferritic) rolling and annealing of low-carbon steels due to the formation of Cr carbides that decrease the amount of available solute carbon in the matrix ⁴⁻⁸).

Concurrent to seeking such improvements, an optimisation of the amount of Cr addition is also mandated in order to minimise the increased alloying costs. For example, a previous study ⁴) showed that a 1.3 wt% Cr addition results in an appreciable improvement in texture and formability. However, there is a significant expense due to the additional alloying that will be ultimately passed onto the consumer. Alternatively, it may be possible to modify the processing schedule and material condition in the as-deformed state such that similar results can be obtained at reduced alloying levels and cost. Therefore, this study investigates the microstructure, bulk texture and solute segregation effects in a low-carbon steel alloyed with 0.78 wt% Cr and subjected to three different processing schedules. Of particular interest is the extent to which C is tied up by the Cr addition (thereby reducing the level of C in solid solution in the matrix) and whether there are any other C segregation effects (in addition to the carbide formation) that need to be accounted for.

2.0 Experimental Procedure

A low-carbon steel containing 0.022C, 0.15Mn, 0.78Cr, 0.035Al, 0.015Ti, and 0.0038N (wt%) was soaked at 930°C for 1 h. After this soak, warm rolling at an average strain rate of 30 s⁻¹ was performed at the EMR-CANMET (Energy, Mines and Resources-Canada Centre for Mineral and Energy Technology) pilot mill in Ottawa, Canada. Three processing schedules were performed: (A) warm rolling at 640°C in two passes to a total reduction of 65%, (B) warm rolling at 580°C in four passes to a total reduction of 80% and, (C) warm rolling at 640°C in two passes to a total reduction of 65% followed by cold rolling to an additional 40% reduction. Upon completion of warm rolling, the samples were water quenched to room temperature for all three processing schedules. The final sheet thicknesses were (A) 3.5 mm, (B) 2.4 mm and, (C) 2.1 mm, respectively.

Standard optical metallography and image analysis techniques were employed to determine: (i) the ferrite grain sizes, (ii) volume fractions of phases and, (iii) amounts of grains containing in-grain shear bands. The shear band morphologies were studied using a JSM 6300F scanning electron microscope (SEM).

A Philips CM20 transmission electron microscope (TEM) operating at 200 kV was used for detailed microstructural characterisation. Both thin foil and carbon replica techniques were applied. The microband and shear band thicknesses were measured by the linear intercept method on five TEM negatives of {111} matrix orientation at magnifications of 15k times.

The atom probe tomography (APT) studies were performed on the Local Electrode Atom Probe (LEAP) at the Oak Ridge National Laboratory. The standard two-stage procedure for atom probe specimen preparation is described elsewhere ⁹⁾. The experiments were performed at a sample

temperature of 60 K with the LEAP operating at a 200 kHz pulse repetition rate and a 0.2 pulse fraction.

The matrix composition was determined in regions that were free of visible solute segregation. The maximum separation envelope method ⁹⁾ was used to assess the extent of solute-enriched regions (such as those associated with clusters, precipitates or atmospheres at dislocations) by distinguishing between atoms that belong to a region of solute concentration and those external to it. The maximum separation distance is estimated by comparing with separation distances between solute atoms in a random solution of the same matrix composition. In this study, this distance (d_{\max}) was 0.85 nm and 2 nm for the clusters and atmospheres, respectively. To eliminate the effect of random fluctuations of the solute in the matrix, the minimum size of solute-enriched regions was assumed to be 20 atoms. The atomic composition of such regions was calculated from the number of atoms of each type forming a cluster or a particle as determined by the maximum separation envelope method. As output of the maximum separation envelope program, the size of the feature is given by the radius of gyration (l_g), which is slightly smaller than what might be considered the actual physical extent. That size might be better represented by the Guinier radius (r_G) given by the following equation ⁹⁾:

$$r_G = \sqrt{\frac{5}{3}} \times l_g \quad (1)$$

A GBC-MMA texture goniometer with a Cu-K α anode and a polycapillary beam enhancer operating at 40 kV and 25 mA was used for the determination of bulk texture. Raw data in the form of incomplete pole figures ($\chi = 0^\circ - 80^\circ$) from the (110), (200), and (211) crystallographic planes was used to calculate the orientation distribution functions (ODFs). Since cubic crystal

and orthorhombic sample symmetries are involved during rolling, the bulk textures are presented between 0° to 90° along both the ϕ_1 and Φ –axes for only the $\phi_2 = 45^\circ$ section.

3.0 Results

3.1 Microstructural Characterisation

The steel microstructure after warm rolling consists predominantly of ferrite and a small volume fraction of pearlite (Figure 1). Greater volume fractions of pearlite were formed after warm rolling at the higher temperature ($\sim 3\%$ at 640°C) than at lower temperature ($\sim 1\%$ at 580°C) (Figures 1(a and b)). As expected with greater rolling reduction, the widths of the grains decreased from $8.7 \pm 0.9 \mu\text{m}$ to $7.2 \pm 0.6 \mu\text{m}$ and $5.7 \pm 0.6 \mu\text{m}$ for the processing schedules A, B and C, respectively. Concurrently, the volume fractions of grains containing in-grain shear bands increased from $\sim 41\%$ after warm rolling (samples A and B) to $\sim 53\%$ (sample C) after additional cold rolling. Two types of shear band morphologies were distinguishable by SEM. The majority of the shear bands were so-called *long* shear bands (Figure 2(a)) which consist of sets of continuous parallel lines crossing entire ferrite grains inclined at $35^\circ \pm 10^\circ$ to the rolling direction. The second type comprised *intense* shear bands which form networks of two intersecting sets of wavy shear bands inclined at $\pm 15^\circ$ to 40° to the rolling direction (Figure 2(b)).

Figure 3 shows the presence of microbands and shear bands in the microstructure of the steel after all processing schedules. Microbands are long thin plate-like features formed on slip planes within individual grains in the rolling direction. The shear bands constitute a form of localized flow within the grains and are inclined at $\sim 35^\circ$ to the rolling plane. TEM analysis revealed that the widths of the microbands and shear bands decreased with greater rolling reduction and lower warm rolling temperatures (Figure 3). The microband and shear band widths decreased from

0.38±0.04 μm to 0.33±0.04 μm and from 0.41±0.06 μm to 0.29±0.04 μm in samples processed according to schedules A and B, respectively. The microband width was further reduced to 0.24±0.02 μm after cold rolling.

The presence of two groups of carbides was also observed in the samples after all three processing schedules: (i) *coarse* carbides of cuboidal and elongated, rounded shapes with widths between 30 and 80 nm and lengths up to 200 nm were predominantly located at grain and microband boundaries (Figures 4(a and b)). (ii) *Fine* spherical and cuboidal carbides 4-30 nm of size (Figures 4(c and d), Table 1) were found within the microbands and shear bands. Combination of indexing of the diffraction patterns and energy-dispersive X-ray spectroscopy (EDXS) indicated the following types of Cr carbides in the matrix: Cr₂₃C₆ (cubic lattice, a = 1.066 nm), Cr₁₉Fe₄C₆ (cubic lattice, a = 1.06 nm) (Figure 4(e)), and Cr₃C₂ (orthorhombic, a = 0.553 nm, b = 0.283 nm, c = 1.147 nm) (Figure 4(d)).

The average diameter of the fine particles tended to be slightly coarser after schedule A (12±1 nm at 640°C) compared to schedule B (11±3 nm at 580°C). However such variations could also be ascribed to the relatively small sampling area employed in the TEM investigation. TEM analysis revealed that the number densities of all particles produced via the three processing schedules were of the same order of magnitude with slightly higher number densities of finer precipitates compared to coarser ones (Table 1). Although the TiC precipitation during warm rolling of the steel might be expected, neither thin foil nor carbon replica TEM examination revealed their presence.

The matrix compositions associated with the three samples were determined by APT and are given in Table 2. Irrespective of the processing schedule adopted, the C level was reduced from the nominal or average alloy value of ~0.1 at% to 0.02-0.04 at%. The Cr content was also reduced from ~0.84 at% to 0.5-0.8 at%. After rolling, the other elements were present at levels similar (within experimental error) to those associated with the nominal steel composition.

APT revealed the presence of Cr-C enriched regions in the ferrite matrix of all the conditions (Figure 5). These regions were estimated from the maximum separation envelope method ⁹⁾ to contain between 20 and 100 atoms and have an average Guinier radius of 1.87 ± 0.24 nm (Table 3). Their compositions range from Cr -rich clusters to clusters containing an approximately 50:50 ratio of Cr and C atoms (Figure 6). A majority of these clusters also contain traces of Fe. However, the method is implemented to aggressively removes the matrix (Fe) atoms. In the samples produced using schedules B and C, some finer clusters containing only Cr atoms were also observed. With an increase in cluster size, the composition typically tends to move towards approximately equal ratios of C and Cr atoms. However in the case of one sample produced via schedule A, a Cr:C ratio as low as 40:60 was also detected. Irrespective of processing schedule, the number densities in all the samples were again of a similar order of magnitude.

The segregation of C atoms to dislocations (i.e. - the formation of Cottrell atmospheres) was also observed by APT (Figure 7). As evident in the C atom map (Figure 7(a)), atmospheres have formed around individual and intersecting dislocations. In most cases, the atmospheres are slightly curved with some straight segments also present. Examples of such segments are shown in Figures 7(b and c) where two perpendicular views are presented of a single atmosphere.

An example of a concentration profile taken perpendicular to the longitudinal direction of an atmosphere is given in Figure 7(d). It shows that the C concentration reaches 4.5 at% at the center of the atmosphere. A more detailed analysis using the maximum separation envelope method showed that such atmospheres have an average Guinier diameter of 4.25 ± 0.41 nm. The numbers of C atoms per equivalent (110), (112) and (222) matrix plane in the observed atmospheres were calculated as described in ¹⁰. These calculations showed approximately 18 ± 12 , 10 ± 7 and 7 ± 5 atoms per equivalent (110), (112) and (222) plane, respectively.

3.2 Bulk Texture Analysis

The $\phi_2 = 45^\circ$ ODF section associated with each of the three processing schedules is shown in Figure 8. In all the ODFs, the rolling textures contain a well-developed ND (or γ) -fibre which runs from $\{111\}\langle 110 \rangle$ to $\{111\}\langle 112 \rangle$ between 60° and 90° along the ϕ_1 -axis and a rolling direction (RD or α) -fibre which runs from $\{001\}\langle 110 \rangle$ to $\{011\}\langle 110 \rangle$ between 0° and 55° along the Φ -axis. As shown in Figure 9, a sample from schedule B displayed slightly stronger γ and α -fibre intensities due to the greater rolling reduction during warm rolling. However, as seen in the sample from schedule C, additional deformation from the cold rolling resulted in an overall weakening of both fibers. The γ -fibre intensities of a sample from schedule C follow the trends the sample from schedule A but are generally weaker. In samples from schedules A and B, the maximum intensity of the α -fibre is located between $\{112\}\langle 110 \rangle$ and $\{111\}\langle 110 \rangle$ whereas there is a minimum in the sample from schedule C in the same region.

4.0 Discussion

Detailed microstructural analysis of the low-carbon steels after the three processing schedules indicates that while the number of grains containing in-grain shear bands increased with the

severity of the deformation, the nature of the in-grain shear bands remained the same. As stated previously, two shear band morphologies were observed and are consistent with previous observations on shear banding in low-carbon steels containing 0.48wt% Cr and subjected to schedule A type processing ⁸⁾. However in that particular study, *short* shear bands were also observed in minor quantities. This type of shear bands was not observed here.

TEM analysis shows that the microband and shear band widths decrease with greater strain. The similarities in the microband widths for the 0.48 wt% Cr ¹¹⁾ and 0.78 wt% Cr (present) steels after 65% warm rolling at 640°C (processing schedule A) indicate that Cr level has no effect on this parameter. Concurrently, the morphologies and types of carbides formed remained the same in both studies. However increasing the amount of Cr did produce a higher number density of Cr carbides. Carbon extraction replicas indicated that the 0.48 wt% Cr steel contained an areal number density of $\sim 10^9 \text{ m}^{-2}$ of carbides, whereas the 0.78 wt% Cr steel had three orders of magnitude higher areal number density of $\sim 10^{12} \text{ m}^{-2}$.

The strain induced precipitates were also slightly coarser in samples warm rolled at 640°C than the samples rolled at 580°C. This difference can be attributed to the greater growth rates associated with the higher processing temperature. The average number density of fine, strain-induced precipitates was higher in the samples after schedule B than in the samples after schedule A, $4 \times 10^{12} \text{ m}^{-2}$ and $0.4 \times 10^{12} \text{ m}^{-2}$ respectively (Table 1). This could be due to the higher magnitude of applied strain in schedule B. However, the large standard deviation associated with the samples from schedule B due to the non-uniform distribution of these precipitates observed in carbon replicas does not permit a statistically valid conclusion. Thus, caution should be exercised when drawing macroscopic conclusions based on the statistical extrapolation of data from the limited sampling volumes used in TEM and APT experiments.

The matrix compositions after all three processing schedules indicate significant depletions in the matrix of both Cr and C. For example, in a sample from schedule A, the concentrations of Cr and C were reduced by factors of 2 and 5, respectively. Although care was taken to analyse the sample volumes free of any visible solute segregation, there was a significant variation in the Cr concentration in different volumes examined after the same processing schedule. This variation can be associated with the formation of very small (<20 atoms) Cr-rich clusters which cannot be unambiguously resolved in atom maps. Although the small clusters can be distinguished using the maximum separation envelope method, they cannot be reliably accounted for during matrix composition analysis due to random solute fluctuations on this length scale. Overall this leads to a small overestimation of the Cr and C levels in the matrix. While the nominal C content in the 0.48wt% Cr steel ¹¹⁾ was 0.17 at% (which is higher than in the present steel (0.1 at% C)), the matrix depletion ratios (the ratio of the nominal C concentration to determined by APT C content) were estimated to be 5.1 and 170 in 0.78 and 0.48wt%Cr steels, respectively. This result is surprising as the 0.78% Cr steel with the higher concentration of the carbide-forming element was expected to undergo even greater C depletion.

A similar trend was observed when the C depletion ratios in low carbon steels with and without additions of 0.48wt% Cr were compared ¹¹⁾. A possible explanation was that there was an increase in C solubility due to the formation of C-Cr dipoles. Formation of such interstitial-substitutional atom pairs in ferritic steels and their contribution to dynamic strain ageing effect was originally proposed in 1970s ^{12, 13)}. This interpretation is also supported by calculations in ¹⁴⁾ which showed that the formation of dipoles increases the C solubility by approximately 10% in low-carbon steels containing 0.5 at% Cr. Thus, an even greater increase in the C solubility can be expected in a 0.78 wt% Cr steel. It worth noting that if this trend is extrapolated to the steel with 1.3wt%Cr, even higher concentrations of carbon in the matrix can be expected; the latter would

decrease the fraction of grains containing in-grain shear bands. This view is supported by the data reported by Barnett⁴⁾ indicating that only 28% of the grains in the 1.3wt%Cr steel contained in-grain shear bands, whereas this number was ~48% in the 0.78wt%Cr steel. At the same time, the 1.3wt%Cr steel displayed a much stronger {111}<112> recrystallisation component⁴⁾ compared to the 0.78wt%Cr steel²¹⁾. It therefore appears that there is an optimum volume fraction of grains containing in-grain shear bands; this fraction can lead under certain conditions to strong γ -fibre formation in recrystallised warm rolled steels via its effect on the mechanism of recrystallisation.

The depletion of Cr and C in the matrix correlates well with the formation of the Cr carbides detected by both TEM and APT. These results show that the reduction in Cr content is greater after processing schedule A than B and is probably due to the higher warm rolling temperature and the associated faster diffusion rate. The carbon was depleted to lower levels after schedules A and C compared to B. As suggested previously, this is more likely to be associated with the higher warm rolling temperature employed for the A and C samples compared to the samples after schedule B. Higher carbon diffusion rates at higher warm rolling temperatures lead to faster carbon segregation at various sinks, such as dislocations, grain boundaries and microbands, and to faster growth of various carbides present in the microstructure.

According to the maximum separation envelope method, the number density of Cr-rich clusters in the 0.78 wt% Cr steel ($1 \times 10^{23} \text{ m}^{-3}$) was approximately two times higher than in the 0.48 wt% Cr steel ($5.1 \times 10^{22} \text{ m}^{-3}$). In contrast to the predominantly C-rich clusters observed in the 0.48 wt% Cr steel, more clusters of Cr-rich composition were seen in the present 0.78 wt% steel. This can be rationalised in terms of the availability of more Cr atoms for precipitate and cluster formation in the 0.78 wt% Cr steel.

The presence of C-rich clusters in the samples from schedule A and not in schedule B could be attributed to the higher density of strain-induced precipitates formed in the latter sample due to the higher magnitude of applied strain. This event may have led to the consumption of more of the available matrix carbon. It is also of interest to note that the cluster compositions in Table 3 are likely to be the precursors for Cr_3C_2 and $\text{Cr}_{19}\text{Fe}_4\text{C}_6$ precipitates.

Comparison of the Cottrell atmospheres indicates that the diameters are greater and that the number of C atoms per equivalent plane is generally higher in the 0.78 wt% Cr steel than those in the 0.48 wt% Cr steel processed via schedule A ¹¹⁾. However their characteristics in the 0.78 wt% Cr steel are in good agreement with previous values reported for C atmospheres forming on dislocations in warm rolled steels with varying composition ¹⁰⁾.

These results have shown that greater additions of Cr to a low-carbon steel do not lead to the further depletion of carbon from the matrix. Furthermore, significant amounts of Cr remained in the solid solution in the matrix in all the samples. This indicates that conventional processing parameters do not allow for the full realisation of the presence of the extra Cr. Although the concentration of solute Cr was increased, other sinks were available for the C atoms, such as atmospheres at dislocations and planar defects (grain boundaries, shear bands and microband boundaries) as well as the possibly higher number of Cr-C dipoles formed. An example of C segregation to the boundaries as detected by APT is presented in Figure 10. Thus, it is evident that Cr competes with other defects for C atoms. The formation of Cr-rich clusters further reduces the number of Cr atoms available for interaction with the C atoms.

The textures observed after warm rolling are similar to those produced in warm and cold rolled low-carbon ^{1, 3, 15, 16)} and interstitial free ^{1, 3, 17)} steels. The slightly higher intensities of the γ - and

α -fibres in the sample of schedule B can be attributed to the higher rolling reduction. However, additional cold rolling in the case of schedule C resulted in a weakening of both fibers.

Barnett *et al.*³⁾ studied warm rolled low carbon steels without Cr additions and concluded that when the strain rate sensitivity parameter is positive, there is a tendency towards sharper texture formation and more uniform microstructure formation¹⁸⁾. Although the strain rate sensitivity parameter was not calculated in the present study, a high positive value is characterised by mobile solute atoms that diffuse to dislocations¹⁸⁾. The latter observation is already seen in the APT investigation (Figure 7) as the C atoms form Cottrell atmospheres at dislocations and provide an additional viscous drag effect at temperature¹⁸⁾. Alternatively when such microstructures are subjected to further reductions via cold rolling, thereby reducing the pinning of dislocations by Cottrell atmospheres, a reduction in rate sensitive parameter causes non-uniform deformation and a proliferation of shear banding. The end effect of such phenomena is an overall weakening of the bulk texture via a scattering of preferred orientations in the as-deformed cold rolled condition.

Although the bulk textures of the warm and cold rolled textures in as-deformed conditions are markedly different, previous studies can be utilised to ascertain their subsequent recrystallisation behaviour. It is well known that good formability in annealed bcc steels is achieved via the retention of a strong ND (or γ) –fiber, which is characterised by the presence of {111} planes parallel to the sheet plane. Analysis of the $\phi_2 = 45^\circ$ ODF sections obtained by Barnett *et al.*³⁾ on low carbon steels indicates sharper and weaker bulk textures after warm and cold rolling, respectively. However, when these samples were subjected to recrystallisation treatments at 700°C, the cold rolled textures retained a strong γ -fiber, whereas the warm rolled textures did not show any γ -fiber orientations¹⁾. In the cold-rolled case, the preservation of the γ -fiber was

ascribed to a combination of shear banding and plane strain deformation conditions whereas the poor performance of the warm rolled samples was due to the formation of {100}<011> -type orientations via strain induced boundary migration ^{1, 18-20}. Deterioration of the γ -fiber strength was produced in the low carbon steels containing 0.48 and 0.78 wt% Cr (previously subjected to warm rolling via processing schedule A) as a result of recrystallisation at 710°C for various time intervals ²¹.

5.0 Conclusions

The microstructures and textures produced in a 0.78 wt% Cr low-carbon steel have been investigated as a function of processing schedule. The results indicate that:

1. The addition of 0.78 wt% Cr increases the volume fraction of Cr carbides by three orders of magnitude compared to the fraction in a 0.48wt% Cr steel. Nevertheless a significant amount of solute Cr remains unused in solid solution in the matrix. Concurrently the level of C in the matrix did not decrease below that observed previously in the 0.48 wt% Cr steel as more Cr-rich clusters and fine carbides were formed.
2. In addition to the changes in the cluster or fine precipitate stoichiometry, it is evident that Cr competes for C atoms in order to form carbides with C segregation to dislocations and boundaries. In effect, this suggests that further increases in Cr content are unlikely to have any significant beneficial effects with respect to removing more carbon from solid solution.
3. It appears that the texture and formability improvements observed by Barnett *et al.* in the steel with 1.3wt%Cr may be due to the optimum number of grains containing in-grain shear bands; the latter may promote strong γ -fibre formation under particular conditions.

4. Rolling reduction via warm followed by cold rolling leads to a greater proliferation of in-grain shear bands but a slightly weaker ND (or γ) fibre in the as-deformed condition. By contrast, warm rolling alone results in sharper textures due to the more positive strain rate sensitivity values.

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